DEVELOPMENT OF A QUANTIFICATION METHOD FOR FOULING DEPOSITS USING PHOSPHORESCENCE

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ABSTRACT

Particulate fouling on structured surfaces is mostly quantified with the integral thermal or mass based fouling resistance. The observed geometries may be structures to improve the heat transfer in heat exchangers (e.g. dimples), cavities in components or more complex geometries. Due to limited accessibility or the requirement of a locally resolved measurement, the existing quantification methods may not be applicable. For this reason, a new method for the quantification for fouling deposits is needed.

In this study dimpled surfaces were evaluate by measuring and comparing the integral thermal and mass based fouling resistance with the local fouling resistance inside and around the dimple. This was carried out online with the Phosphorescent Fouling Quantification method developed for this purpose, using phosphorescent particles to quantify the deposited mass. The mass based fouling resistance can be calculated using computer aided image analysis.

The measurements for the evaluation are conducted on dimpled surfaces. On these surfaces, a characteristic fouling pattern was observed. A reduced surface coverage and thus lower fouling resistances downstream of the dimple compared to a plain surface could be seen. These results confirm earlier findings, suggesting an advantage of dimpled surfaces against other surface structures with respect to thermo-hydraulic efficiency as well as reduced fouling. Thus, the Phosphorescent Fouling Quantification method provides the possibility to calculate values for local fouling resistances on structured surfaces and thus an optimization of surface structures to minimize fouling propensity.

INTRODUCTION

Particulate fouling on structured surfaces

Particulate fouling on heat exchanger surfaces is one of the main reasons for efficiency problems and its prevention is a major challenge in processing industry [1]. The decrease of heat transfer due to fouling often leads to overdesign and higher energy and maintenance costs [2]. Therefore, for the evaluation of the long-term efficiency of heat exchangers, the fouling propensity must be taken into account in addition to the thermo-hydraulic efficiency which is based on the ratio of heat transfer to flow resistance. Heat exchangers with dimpled surfaces have a comparatively high thermohydraulic efficiency. They are used for cooling with river water, where the fouling probability is high due to the suspended particles, e.g. sand or corrosion products.

The mechanisms of particulate fouling on plate heat exchangers are described among others by Grandgeorge et al. [3]. Suspended particles are transported from the bulk fluid to the wall surface due to inertia forces or diffusion and adhere to it. The adhesion of particles to the surface is mainly induced by van der Waals and electrostatic forces [4]. The formation of fouling layers occurs when the particle deposition rate is higher than the removal rate. The opposite case of a higher removal rate is used technically to clean soiled surfaces [5]. Therefore, higher shear stresses can be achieved by increasing the fluid velocity and by using surface structuring. For the investigation of the effect of structuring on the various mechanisms - deposition, suppression or removal - the fouling layers have to be quantified on a local basis.

Quantification of particulate fouling

In most studies the thermal fouling resistance is used for the quantification of particulate fouling on heat exchanging surfaces, e.g. [6]. Thereby the fouling resistance is measured either integrally or locally with the corresponding temperatures. In both cases, the accuracy of the method is highly dependent on the measurement technology. Furthermore a heat transferring surface is always necessary, since the heat flux is required for the calculation of the overall heat transfer coefficient and therefore of the thermal fouling resistance.

Additionally, particle fouling also occurs without the presence of a temperature gradient, e.g. in piping systems without accompanied heat transfer. For isothermal processes with the formation of fouling layers the mass based fouling resistance is applicable. Besides material parameters the mass of the fouling layer has to be determined here. This method is invasive in most cases and destroys the fouling layer due to drying or extraction of the layer. Also the fouling process has to be interrupted for analysis. So the result is highly influenced by the measurement procedure.

Thermal fouling resistance

The integral thermal fouling resistance is based on temperature measurement of the inlet and outlet streams of product and heating fluid in the flow channel. First of all the overall heat transfer coefficient for the clean channel k_0 is calculated with the heat flow \dot{Q} between the two streams, the heat transfer area A and the logarithmic mean temperature difference ΔT_m of the integrally measured temperatures:

$$k_0 = \frac{\dot{Q}}{A\Delta T_m} \tag{1}$$

The heat flow through the wall \dot{Q} is calculated either from the heat emitted by the heating fluid or the heat absorbed by the product stream. The temperature difference ΔT_i in eq. (2) is determined between the inlet and outlet flow of the channel. The temperature dependent data for the density ρ which is necessary to calculate the mass flow \dot{m}_i and the heat capacity c_p are taken from literature according to the arithmetic mean temperature of the fluid. It is assumed, that the suspended particles in the used concentration of 0.2 g/L do not affect the properties of the water. The calculated values of the heat flows of the product and heating side should be identical if no heat loss occurs.

$$\dot{Q}_i = \dot{m}_i c_{p,i} \Delta T_i \tag{2}$$

During the fouling experiment the timedependent overall heat transfer coefficient is determined too. It is now influenced by the fouling process and defines the heat transfer in the heat exchanger including the influence of the deposited fouling layer. The value of the new overall heat transfer coefficient k_f is smaller than k_0 due to additional thermal resistance of the fouling layer, the so called thermal fouling resistance $R_{f,th}$.

$$R_{f,th} = \frac{1}{k_f} - \frac{1}{k_0} \tag{3}$$

Mass based fouling resistance

The parameters for the mass based fouling resistance $R_{f,m}$ are achieved with an invasive method as described by Deponte et al. [7]. The specific mass m_f of the deposited particles is determined by extraction of the dried fouling layer from the plate with adhesive tape. Thereby a tape with the geometry of 18 mm × 18 mm is used. Two tapes are

placed on the surface of the test plate right upstream and downstream of the dimple. The particles adhering to the adhesive tape are weighted with an accuracy of 0.1 mg which equals an accuracy of the specific mass m_f of 0.03 g/m².

The equation for the mass based fouling resistance results from eq. (4) with the thermal conducticity $\lambda_{f,tot}$ and the thickness x_f of the overall fouling layer [8]:

$$R_{f,m} = \frac{x_f}{\lambda_{f,tot}} \tag{4}$$

The overall thermal conductivity $\lambda_{f,tot}$ is defined by the thermal conductivity of the fouling material λ_f and the thermal conductivity of the fluid, in this case water λ_w , which fills the cavities between the particles and is part of the stagnant layer [9]. The proportion of the involved thermal conductivities results from the void fraction ϵ which defines the free volume inside the deposit. For the calculation of the void fraction a defined volume of the fouling layer is observed, the cell volume V_{cell} . Thereby the occupied volume is calculated with the number N_p and volume V_p of the particles in the observed cell The occupied volume of a cell is defined in eq. (5).

$$(1-\varepsilon) = \frac{N_p V_p}{V_{cell}} \tag{5}$$

The value for the packing factor $(1 - \varepsilon)$ has to be assumed in order to calculate the mass based fouling resistance. Due to their sphericity the particles will arrange like sphere packings. This limits the possible values for the packing factor to the range of $(1 - \varepsilon) = 0.52$ (simple cubic) to $(1 - \varepsilon) = 0.74$ (face-centered cubic). A random packing generally has a packing factor of around $(1 - \varepsilon) = 0.64$ [10]. For the further evaluation of fouling resistances in this study this value is used for the calculation of $R_{f,pfq}$ and $R_{f,m}$.

In the proportion given by the void fraction of a random packing, considered as homogeneous, the thermal conductivities are described mathematically as series-connected resistances to calculate the overall thermal conductivity $\lambda_{f,tot}$ [11].

$$\frac{1}{\lambda_{f,tot}} = \frac{1-\varepsilon}{\lambda_f} + \frac{\varepsilon}{\lambda_w}$$
(6)

Thus, the mass based fouling resistance related to the height of the fouling layer follows from inserting eq. (6) into eq. (4).

$$R_{f,m} = x_f * \left(\frac{1-\varepsilon}{\lambda_f} + \frac{\varepsilon}{\lambda_w}\right) \tag{7}$$

The mean height x_f of the fouling layer, necessary for the determination of the mass based fouling resistance, is calculated with the specific mass m_f and density of the deposited particles ρ_f . The value is corrected with the void fraction ϵ .

$$x_f = \frac{m}{\rho_f(1-\varepsilon)} \tag{8}$$

The mass based fouling resistance follows from combining eq. (8) and eq. (7).

$$R_{f,m} = \frac{m}{\rho_f(1-\varepsilon)} * \left(\frac{1-\varepsilon}{\lambda_f} + \frac{\varepsilon}{\lambda_w}\right)$$
(9)

This method is invasive and destroys the fouling layer and is therefore the last applied method. This analytical method differs from the others, because it can only be used after completion of the experiment. With the PFQ methode and the method for the thermal fouling resistance it is possible to obtain results online during the experiment.

METHODS

Phosphorescent Fouling Quantification (PFQ) method

In this study, a new method is developed for the evaluation of heat transfer surfaces, the Phosphorescent Fouling Quantification (PFQ). This method quantifies the deposited mass by using phosphorescent particles as fouling material. This is a non-invasive method for the online determination of local mass based fouling resistances.

The principle of the PFQ method is based in the Local Phosphorescence Detection (LPD) method which was first introduced by Schöler et al. [12]. The LPD was used for the evaluation of the local cleaning efficiency of pulsed flow cleaning procedures for Cleaning in Place (CIP) processes in the food industry. Phosphorescent crystalline zinc sulfide was integrated in the applied fouling layer as a tracer. The cleaning progression was monitored optically by detection of the emitting light of the tracer and was assessed with the resulting coverage of the surface. This method was evaluated against other methods for studying CIP mechanisms by Gordon et al. [13] and was assessed as a suitable method for the measuring of the cleaning time.

The PFQ method is using phosphorescent particles which are dispersed in an aqueous suspension with a concentration of c = 0.2 g/l. Simultaneously the tracer particles are acting as fouling material. The particles used are copperdoped zinc sulfide crystals (Lumilux green SN-F5, Honeywell) which exhibit a strong phosphorescence and provide a high sphericity of $\psi = 0.88$. Fig. 1 shows an image of the particles taken with a scanning electron microscope.



Fig. 1. Image of the Lumilux green SN-F5 particles taken with scanning electron microscope.

The particles have a narrow particle size distribution with a median diameter of $d_{p,50} = 1.14 \ \mu$ m, see Fig. 2. For the determination of the mass based fouling resistance, the density of the particles is necessary, which is $\rho_f = 4,090 \ \text{kg/m}^3$. The copper activator leads to a green long-time afterglow of the material after activation with visible light. This emitting light is detected with a camera in a suitable housing which excludes ambient light. The images can be taken during the fouling process through the glas windows in the lid of the channel without interruption or influencing the particulate flow and deposition. Therefore the fouling progression can be observed over time.



Fig. 2. Cumulative particle size distribution of Lumilux green SN-F5.

The captured images are processed using an analysis methode created in Matlab® (Version R2016a 9.0.0.341360, MathWorks, 2016). This procedure is shown schematically in Fig. 3. The true color images are converted from the rgb color space into a grayscale intensity image by eliminating the information for hue and saturation. The luminance is retained and its value for each pixel of the image is saved in a matrix. Therefore, the matrix has the same dimensions as the resolution of the image. The luminescence value is proportional to the amount of deposited particles on the surface. The mathematical

correlation of luminescence and height of fouling layer is deducted for each image from the calibration with local height measurements performed with a digital microscope. The results are then displayed in a scaled heat map.



Fig. 3. Image processing procedure of the Phosphorescent Fouling Quantification (PFQ).

With the use of the physical data of the fouling layer material the height of fouling layer x_f can be converted into the mass based fouling resistance $R_{f,m}$. In order to avoid confusion with other methods, the mass based fouling resistance measured with the PFQ is indicated as $R_{f,pfq}$.

$$R_{f,pfq} = x_f * \left(\frac{1-\varepsilon}{\lambda_f} + \frac{\varepsilon}{\lambda_w}\right) \tag{10}$$

The values for the temperature dependend thermal conductivity of water is taken from [14]. The deposited particles have a thermal conducticity of $\lambda_f = 27.2$ W/(m K). It is assumed that the thermal conductivity of the solid material is not temperature dependent in the given temperature range.

The PFQ method is evaluated with experiments on particulate fouling on dimpled surfaces. The mass based fouling resistance achieved with the PFQ is validated with the values of the mass based fouling resistance which are measured at the end of each experiment. Furthermore the time dependent fouling behaviour expressed with the course of the thermal fouling resistance is compared with the online measured fouling resistances of the PFQ method.

Experimental setup

The experimental setup of the test rig used for the investigations of the fouling behaviour of particles in aqueous suspensions on structrured surfaces is described in detail by Deponte et al. [7]. The fouling experiments where conducted in a flow channel with a rectangular cross section of 4 mm height \times 18 mm width resulting in a hydraulic diameter of d_h = 6.55 mm. The length of the channel is 520 mm. Glass windows are inserted in the lid for optical accessibility of the test plate. The setting of the process parameters used for the experiments is shown in table 1.

Table 1 Setting of process parameters used for the experiments.

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Parameter	Symbol	Setting	Unit
Fluid velocity	u _{Susp.}	1	m/s
Reynolds number	Re _{Susp.}	8,200	-
Temperature	T _{Susp.}	30	°C
Time	t	1	h

A second channel supplies the heating fluid water in countercurrent flow. It enters at 50 °C with a fluid velocity of 1.7 m/s. The heating channel is located 2 mm under the test plate and has the same rectangular cross section as the flow channel. The cross section of the channels and the test plate are shown in Fig. 4.



Fig. 4. Cross section of flow and heating channel with the test plate in between.

Test plates

Dimpled surfaces were used to evaluate the PFQ method. They provide local pattern of the fouling deposit due to a self-cleaning effect, which was described by Kasper et al. [15]. Kasper et al. observed a trail downstream the dimple with a decreased particle deposit on the surface inside the trail in comparison to the area upstream of the dimple, see Fig. 5. The observations lead to the hypothesis that dimpled surfaces in heat exchangers are advantageous over other surface structures with respect to reduced fouling propensity.



Fig. 5. Characteristic fouling pattern on a dimpled surface with the visible trail downstream of the dimple; fluid flow is in x-direction [15].

The self-cleaning effect is numerically and experimentally visualized on the surface downstream of the dimple. The first quantification of the self-cleaning effect of dimples was shown by Deponte et al. [7] by measuring the decrease of coverage of the surface.

In addition to the reduced fouling propensity, the thermo-hydraulic efficiency of dimpled surfaces is enhanced in comparison to a planar plate [16]. The thermo-hydraulic efficiency is defined as the ratio of heat transfer to flow resistance, which is used for the evaluation of structured surfaces in heat exchangers with and without dimples [17]. Dimples with the ratio of dimple depth t_D to diameter D of $t_D/D = 0.26$ were identified numerically as the best geometry with respect to thermo-hydraulic efficiency [18].

The test plates used for the experiments are 500 mm in length and are made of bright stainless steel (1.4301). The plate has one single dimple with the ratio of dimple depth to diameter of $t_D/D = 0.26$, positioned after 100 mm in flow direction on the plate. It is designated in the following text as S26B.

For a comprehensive approach to evaluate structured surfaces values for the fouling resistance should be considered. But the methods for measuring the thermal or mass based fouling resistance are not applicable inside the cavity of the dimple. This is where the PFQ method is used.

RESULTS AND DISCUSSION

Evaluation of the PFQ method

The PFQ method is evaluated on dimpled surfaces. It is hypothesized, that spherical dimples suppress the deposition of particles from a liquid stream or even induce a permanent cleaning of the surface. In addition to the numerical studies by Kasper et al. [15] and the experimental studies by Deponte et al. [7], the self-cleaning effect can be observed here with locally resolved fouling resistances. Furthermore the PFQ method provides insights into the dimple itself. The surface of the dimple has not yet been examined experimentally.



Fig. 6. Local fouling resistances around and inside the dimple; fluid flow is in x direction.

Fig. 6 shows the local distribution of the fouling resistance matrix R_{f,pfq} on the surface of the test plate inside and around the dimple. The characteristic fouling pattern, observed in studies before, is visible and quantified by the PFQ results. The area downstream of the dimple is less covered with particles than the area upstream of the dimple. For the local analysis of the fouling layer distribution parts of the R_{f,pfq} matrix are extracted. Fig. 7 shows an elevated fouling resistance on the outer edge of the plate and a constant value on the central surface upstream of the dimple. This effect is described by the boundary condition of the flow. Due to decreasing flow velocity in the boundary layer near the wall the propensity of particle deposition is increased. The reduction of the value at the far outer edge of the plate shows the small area in which the plate is clamped in the channel. The core flow has a constant velocity in a turbulent flow regime, thus the fouling resistance of about $4.8 \times 10^{-7} \text{ m}^2\text{K/W}$ is constant too in this area.



Fig. 7. Fouling resistance upstream of the dimple (mean values of pixels-lines no. 125 to 130 (Fig. 6) in x-position).

The PFQ method allows for local investigation of the fouling resistance inside the dimple. Fig. 8 shows the cross section in flow direction through the center from front to end of the dimple. The highest fouling resistance is measured at the front rim of the dimple. On the further surface of the dimple the deposited mass is slightly reduced compared to the area upstream the dimple. The mean fouling resistance in this area is about 4.4×10^{-7} m²K/W.



Fig 8. Cross section in flow direction from front to end through the center line of the dimple.

The area of a pixel projected to the plate surface is $0.03 \text{ mm} \times 0.03 \text{ mm}$. The results show that the local differentiation of the distribution of fouling resistances is very accurate so the self-cleaning effect of the dimples can be quantified. If nessecary the resolution of the PFQ method can easily be increased by using other lenses or a camera with a larger sensor.

Comparison of methods

For the comparison of the three applied methods each fouling resistance is measured after 60 min on the test plate with one dimple S26B. The mean value of five measurements for each method is shown in Fig. 9. The thermal fouling resistance is measured integrally over the whole plate. The other two methods are applied upstream of the dimple so the influence of the dimple is not considered in this value.



Fig. 9. Comparison of the measured fouling resistance with different methods.

All three methods provide comparable results, while the standard deviations are clearly different. The thin fouling layers decrease the overall heat transfer coefficient just slightly and the change in the resulting temperature difference is low. So the uncertainty in the thermal fouling resistance $R_{f,th}$ is high. However, the mean value of the five measurements is comparable with the other methods.

While the application of all three methods is straightforward, the analytical effort varies. The thermal fouling resistance can be calculated online with the direct output of the process control system. The PFQ method needs images captured during the process which have to be evaluated offline. This is done with a MatLab® procedure. The mass based method requires the most analytical effort after the conduction of the experiment.

Next to the analytical effort, the instrumental effort has to be considered. Both the PFQ and the mass based method do not need a heat transferring surface. The provided heating channel is just necessary for the determination of the thermal fouling resistance. In contrast to the other methods, the PFQ has certain requirements for the fouling material. It is essential, that the particles provide detectable phosphorescence. For fundamental studies of particulate fouling phosphorescent particles of various sizes and densities are available. The investigation of other fouling mechanisms like crystalline or bio fouling requires additional tracer particles, if no phosphorescence is provided by the system itself. Additionally, the PFQ may not be suitable for very thick fouling layers. If several layers are on top of each other the emitted light of the lower layers may not be detected by the camera.

The application of the PFQ needs a calibration for each fouling problem to calculate fouling resistances. Additionally the maximal detectable height of the layer and light intensity must be known to be sure that the emitting light of all layers is detected.

Time resolved fouling progression

After the experiment, a fouling layer on each test plate is clearly visible. The observation through the glass windows in the lid of the channel suggests that the fouling layer is growing at a constant rate. This assumption is supported by the course of the measured fouling resistance. The progression of the thermal fouling resistance and the fouling resistance measured with the PFQ are shown in Fig. 10 for the test plate S26B during a single fouling experiment. The PFQ fouling resistance is measured online through the windows of the lid of the channel and is the mean value for an area of 80 mm \times 18 mm on the test plate with the dimple in the middle of the image.



Fig. 10. Comparison of the progression of fouling resistance of a single experiment on S26B measured thermally and with PFQ.

The evaluation of the two methods shows a comparable course of the increase in fouling resistance over time. On average the thermal fouling resistance is slightly higher than $R_{f,pfq}$ which is due to the area used for the analysis. The integral thermal fouling resistance refers to the total surface of the plate, so the area influenced by the dimple is

comparatively smaller. In this area the self-cleaning takes place, the local quantification is described in the next section.

An asymptotic behavior of the fouling resistance is assumed but cannot be clearly verified in this experiment of one hour. The fouling resistance still seems to increase, although at a lower rate than at the beginning of the experiment. For a final evaluation, the measuring period must be increased.

Locally resolved fouling resistance

Even if the determination of the mass based fouling resistance is invasive, it is the only direct measuring method for fouling deposits. It does not need a tracer nor a heat transferring surface for the determination of the fouling resistance.

The particulate deposition can be resolved locally with the mass based fouling resistance and also with the fouling PFQ method e.g. upstream and downstream of the dimple to confirm the statements of previous studies which claim a self-cleaning of dimpled surfaces [7, 15]. The results of the mass based fouling resistance and the fouling resistance measured with the PFQ up- and downstream of the dimple are shown in Fig. 11.





The expected lower fouling resistance downstream of the dimple due to their self-cleaning effect can be measured with both methods. Referred to the fouling resistance upstream of the dimple the selfcleaning, respectively the reduction of the fouling resistance, is 33.3 % for the mass based method and 46.9 % for the PFQ method. The underestimation of the mass based Fouling resistance can be explained by a uncomplete extraction of the fouling layer with the adhesiv tape. This is especially the case by the measurement of thick layers.

The higher precision of the PFQ method is due to the measuring technique: While the observed area of the mass based method is limited to the minimum measurable and weighable size of the adhesiv tape, the PFQ allows even smaller local distinctions limited to the resolution of the camera sensor. Furthermore the application of adhesive tape on structured surfaces may not be possible due to shape and size of the structuring. This limitation does not apply to the PFQ method. Even in cavities difficult to access fouling resistances are possible to measure if optical accessibility is available.

CONCLUSION

It could be shown that the PFQ method is well applicable for the evaluation of the local fouling behavior of structured surfaces such as dimples. Even though the thermal, the mass based and the fouling resistance measured with the PFQ method are applicable for the measurement of the fouling resistance, the PFQ has some advantageous: The measurement can be carried out online with a high local resolution. However, it inherently depends on the emitting light of the fouling layer. The Phosphorescent Fouling Quantification does not need a heat transferring surface for the measuring of fouling resistances like the thermally measured fouling resistance. Furthermore the PFQ method is not invasive like the mass based fouling resistance. Thus, the fouling process is not interrupted while the measurement is obtaining results online. This also has the advantage that the fouling resistances can be calculated automatically during the process.

The suitability of the method for fundamental research was shown in this study. Further investigations with different dimple geometry and multiple dimples in a row on the test plate will be conducted with this method. Furthermore the method will be validated with the results from numerical simulation of particle deposition on dimpled surfaces.

NOMENCLATURE

Symbols

- c concentration; g/l
- c_p heat capacity; J/(kg K)
- D dimples diameter; m
- d diameter; m
- k overall heat transfer coefficient; W/(m² K)
- m specific mass; kg/m²
- *m* mass flow; kg/s
- N number; -
- \dot{Q} heat flow; W
- R_f fouling resistance; m² K/W
- Re Reynolds number; -
- T temperature; K
- t time; s
- $t_{\rm D}$ dimples depth; m
- u fluid velocity; m/s
- V volume; m³
- x laver thickness: m
- Δ difference: -
- ε void fraction: -
- λ thermal conductivity; W/(m K)

- ρ density; kg/m³
- ψ sphericity; -

Subscripts

g

- f fouling material, with fouling
- h hydraulic
- m mass based, logarithmic mean
- p particle
- p,50 mean particle value
- pfq based on PFQ method
- Susp. suspension
- th thermal
- tot overall fouling layer
- w water

Abbreviations

LPD Local Phosphorescence Detection

PFQ Phosphorescent Fouling Quantification

S26B Test plate with one dimple, $t_D/D = 0.26$

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